

**Original Research Article**  
**Solvent free synthesis of 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide derivatives as Novel antimicrobial and antitumor agents**

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## ABSTRACT

In pursuit of designing novel chemical entities with antitumor and antimicrobial activities, 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide derivatives have been synthesized as a scaffold of a series of amine derivatives, these analogs were on reductive amination with solvent free condition. All the synthesized compounds were assessed for antibacterial, antifungal, and antitumor activities against standard strains. The compounds 3a, 3b and 3g showed highest degree of inhibition against *A. Flavus* and compounds 3a, 3b, 3d and 3h Showed highest degree of inhibition against *C. albicans*, compounds 3a,3e and 3g has shown the encouraging antibacterial activity results against *E.coli* and compounds 3a, 3b and 3h exhibited a promising activity against *B. subtilis*. Compounds 3a, 3b and 3c exhibited promising antitumor activity.

*Keywords: Benzoxazole, Solvent free condition. Antitumor, antimicrobial.*

## 1.INTRODUCTION

Benzoxazole skeleton have been found in numerous biological and pharmaceutical substances and which showed wide spectrum of pharmaceutical properties. In support for this study, substituted benzoxazoles showed various activities including antineoplastic, antitubercular, anthelmintic, and antimicrobial, anti-HIV, antifungal, antiprotozoal [1-6]. The transformation of amines through different aldehydes is an important method in synthetic chemistry because of their benefit as intermediates for the preparation of agrochemicals and pharmaceuticals [7-8]. It is thought worthwhile to carry out the synthesis of title compounds. In point of view to obtain some new chemical entities with together effective pharmacophores in a distinct molecular framework for the increased biological activities.

In account of the aforementioned information and with the extension of the former investigations on benzoxazole skeleton and its pharmacological activities [9-15], the preparation of a new series of compounds (Scheme-2) have been achieved. The present work sheds light on the cost effective solvent free synthetic route for the synthesis of sequence of 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide derivatives. The synthesized derivatives have been subjected to biological activities, which displayed significant activities against all the strains.

## 2.METHODOLOGY

### 2.1 Materials and Methods

By taking the sample of compounds in a glass capillary tube being sealed at one end, melting points were determined in an electrically heated apparatus, which are uncorrected. The purity and reaction progress of all the compounds were checked by TLC on silica gel plates using n-Hexane, ethyl acetate solvent system and spots located by UV and iodine chamber. All the chemicals used in this were purchased from Sigma-Aldrich and SD Fine. IR spectra were recorded using KBr pellets on a Perkin Elmer Spectrophotometer. <sup>1</sup>H-NMR spectra on Agilent 400 MHz Spectrophotometer and chemical shifts were expressed as ppm and TMS as internal standard. Mass spectra were recorded on Xevo G2-XS Qtof.

### **2.1.1 Synthesis of 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide (2)**

Methyl-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetate (1 g, 0.0039 mol, 1eq) was added to ethanol (50 mL) and hydrazine hydrate (0.22 mL, 0.0047 mol, 1.2 eq) was added slowly. The reaction mixture was stirred under reflux in absolute alcohol for 4 hours. Progress of the reaction was confirmed by TLC (Chloroform: Methanol in 7:3 ratio). Reaction mixture was then poured to ice water to obtain compound 2. [16-20].

White solid; yield (90%), MP. (184-186°C); IR (KBr,  $\text{cm}^{-1}$ ): 1592 (C=N), 3330 (N-H); <sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$ : 2.4 (s, 3H), 4.02 (s, 2H), 4.32 (br, 2H, NH<sub>2</sub>), 7.07-7.48 (m, 3H), 9.4 (br, 1H, NH) (D<sub>2</sub>O exchangeable); <sup>13</sup>C NMR (DMSO):  $\delta$  21 (CH<sub>3</sub>), 39 (CH<sub>2</sub>), 110 – 135 (aromatic carbons), 146 (C=N), 166 (C=O); MS (m/z): 237 (M<sup>+</sup>).

### **2.1.2 General procedure for the synthesis of 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide derivatives 3(a-h)**

A 2-[(5-Methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide amine (1mmol) was ground with a substituted aldehyde (1 mmol) for 15 to 20 minutes in an agate mortar and pestle at room temperature (25°C) under solvent free conditions. To the resulting mixture 1:1 ratio of Sodium borohydride and boric acid and then the mixture was added ground for 20-30 minutes until TLC showed complete disappearance of the aldehyde. The reaction mixture was washed with water and further purified by recrystallization with ethanol. [21-25]

### **2.1.3 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]-N'-(thiophen-2-ylmethyl)acetohydrazide (3a)**

Grey colour solid, yield (75 %) MP (235-236°C); IR( $\text{cm}^{-1}$ ): 1166 (C-N str.); <sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$ : 2.33 (s, 3H), 4.22 (s, 1H), 4.6 (s, 2H), 7.04 (s, 2H), 7.30 - 7.64 (Ar-H's), 11.80 (s, 1H); <sup>13</sup>C NMR (DMSO)  $\delta$ : 21.3 (CH<sub>3</sub>), 34.1 (CH<sub>2</sub>), 35.00 (CH<sub>2</sub>), 110-150 (Aromatic carbons), MS (m/z): 333 (M<sup>+</sup>)

### **2.1.4 N'-(furan-2-ylmethyl)-2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide (3b)**

Grey colour solid, yield (73 %), MP (250-253°C); IR( $\text{cm}^{-1}$ ): 1156 (C-N str.); <sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$ : 2.33 (s, 3H), 4.22 (s, 1H), 4.6 (s, 2H), 7.04 (s, 2H), 7.30 - 7.64 (Ar-H's), 11.80 (s, 1H); <sup>13</sup>C NMR (DMSO)  $\delta$ : 21.3 (CH<sub>3</sub>), 34.1 (CH<sub>2</sub>), 35.00 (CH<sub>2</sub>), 110-150 (Aromatic carbons) MS (m/z): 317 (M<sup>+</sup>)

### **2.1.5 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]-N'-(2-nitrobenzyl)acetohydrazide (3c)**

Grey colour solid, yield (75 %), MP (296-298°C); IR( $\text{cm}^{-1}$ ): 1156 (C-N str.); <sup>1</sup>H NMR (400 MHz, DMSO)  $\delta$ : 2.41 (s, 3H), 3.98 (s, 1H), 4.6 (s, 2H), 7.04 (s, 2H), 7.30 - 7.64 (Ar-H's), 11.66

(s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.42 (CH<sub>3</sub>), 34.01 (CH<sub>2</sub>), 35.00 (CH<sub>2</sub>), 109-150 (Aromatic carbons) ; MS (m/z): 372 (M<sup>+</sup>)

### **2.1.6 N'-(4-chlorobenzyl)-2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide (3d)**

Grey colour solid, yield (74 %), MP (280-282°C); IR(cm<sup>-1</sup>): 1152 (C-N str.);<sup>1</sup>H NMR (400 MH, DMSO)  $\delta$ : 2.45 (s,3H), 3.97 (s,1H), 4.62 (s, 2H), 7.02 (s, 2H), 7.30 - 7.64 (Ar-H's),11.66 (s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.42 (CH<sub>3</sub>), 29.01 (CH<sub>2</sub>), 34.00 (CH<sub>2</sub>), 109-147 (Aromatic carbons), 183.72 (C=O); MS (m/z): 362 (M<sup>+</sup>).

### **2.1.7 N'-benzyl-2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide (3e)**

Grey colour solid, yield (77 %), MP (230-232°C); IR(cm<sup>-1</sup>): 1142 (C-N str.);<sup>1</sup>H NMR (400 MH, DMSO)  $\delta$ : 2.41 (s,3H), 3.97 (s,1H), 4.64 (s, 2H), 7.01 (s, 2H), 7.11 - 7.71 (Ar-H's),11.07 (s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.55 (CH<sub>3</sub>), 34.17 (CH<sub>2</sub>), 34.57 (CH<sub>2</sub>), 109-150 (Aromatic carbons), 169.37 (C=O); MS (m/z): 327 (M<sup>+</sup>).

### **2.1.8 N'-(2-chlorobenzyl)-2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide (3f)**

Grey colour solid, yield (78 %), MP (285-287°C) ; IR(cm<sup>-1</sup>): 1164 (C-N str.);<sup>1</sup>H NMR (400 MH, DMSO)  $\delta$ : 2.39 (s,3H), 4.25 (s,1H), 4.68 (s, 2H), 7.10 (s, 2H), 7.11 - 7.71 (Ar-H's),11.92 (s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.38 (CH<sub>3</sub>), 34.98 (CH<sub>2</sub>), 35.17 (CH<sub>2</sub>), 110-164 (Aromatic carbons), 168.69 (C=O); MS (m/z): 361 (M<sup>+</sup>).

### **2.1.9 N'-(2-bromobenzyl)-2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]acetohydrazide (3g)**

Light brown colour solid, yield (80%), MP (298-299°C); IR(cm<sup>-1</sup>): 1144 (C-N str.);<sup>1</sup>H NMR (400 MH, DMSO)  $\delta$ : 2.45 (s,3H), 3.92 (s,1H), 4.61 (s, 2H), 6.98 (s, 2H), 7.12 – 8.0 (Ar-H's), 11.18 (s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.39 (CH<sub>3</sub>), 34.16 (CH<sub>2</sub>), 34.27 (CH<sub>2</sub>), 110-164 (Aromatic carbons), 169 (C=O); MS (m/z): 406 (M<sup>+</sup>).

### **2.1.10 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl]-N'-(4-nitrobenzyl)acetohydrazide (3h)**

Grey colour solid, yield (71 %), MP (280-281°C); IR(cm<sup>-1</sup>): 1174 (C-N str.);<sup>1</sup>H NMR (400 MH, DMSO)  $\delta$ : 2.31 (s,3H), 4.12 (s,1H), 4.68 (s, 2H), 6.74 (s, 2H), 7.08 – 8.0 (Ar-H's), 12.02 (s, 1H);<sup>13</sup>C NMR (DMSO)  $\delta$  : 21.37 (CH<sub>3</sub>), 34.90 (CH<sub>2</sub>), 39.36 (CH<sub>2</sub>), 110-164 (Aromatic carbons), 168 (C=O); MS (m/z): 372 (M<sup>+</sup>).

## **2.2 Biological Activities**

### **2.2.1 Antibacterial Activity**

The newly synthesized 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide derivatives were tested for antibacterial activity against bacterial strains, *Escherichia coli* (ATTC-8739), *Bacillus subtilis* (ATTC-6633) by agar well diffusion method [26-27]. The 24 hr old Mueller-Hinton broth culture of test bacteria were swabbed on sterile Mueller-Hint on agar plates using sterile cotton swab followed by punching wells of 6 mm with the help of sterile cork borer. The standard drug (Streptomycin 1mg/mL of sterile distilled water), compounds 3(a-h) (25mg/mL of 10% DMSO), and control (10% DMSO) were added to the respectively label dwells. The plates were allowed to stand for 30 minutes and were incubated at 37°C for 24 hr in upright position and the zone of inhibition was recorded and tabulated in Table 2. And compounds 3a,3e and 3g have shown the very good activity against *E.coli* and compounds 3a, 3b and 3h showed considerable activity against *B. subtilis*.

### **2.2.2 Antifungal Activity**

The Compounds 3(a-h) were screened for anti-fungal activity against fungal *A. flavus* and *C. albicans* strain with fluconazole as a standard drug respectively. The compounds 3a, 3b and 3g Showed inhibition against *A. flavus* when compared with the standard drug fluconazole. Compounds 3a, 3b, 3d and 3h showed inhibition against *C. albicans* when compared with the standard drug fluconazole [28-29].

### **2.2.3 Antitumor activity**

The antitumor activity against HepG2, human liver cancer cell lines were estimated using the 3-[4,5-dimethyl-2-thiazolyl)-2,5-diphenyl-2H-tetrazolium bromide (MTT) assay, Cells were dispensed in a 96-well sterile microplate ( $5 \times 10^4$  cells/well) and incubated at 37 °C with series of different concentrations in DMSO, of 3(a-h) compounds for 48 h in a serum-free medium prior to the MTT assay. After incubation, media were carefully removed, and 40  $\mu$ L of MTT (2.5 mg/mL) was added to each well and then incubated for an additional 4 h. The purple formazan dye crystals were solubilised by the addition of 200  $\mu$ L of DMSO. The absorbance was measured at 570 nm using multi-Mode micro plate reader analysed using Megalen software. The relative cell viability was expressed as the mean percentage of viable cells compared to the untreated control cells. All experiments were conducted in triplicate and repeated in three different days. All the values were represented as mean  $\pm$  SD and IC50s were determined [30-32].

**Table 1. Antibacterial activity**

Compounds	Bacteria			
	<i>E. coli</i>		<i>B. subtilis</i>	
	25 mg/mL	50 mg/mL	25 mg/mL	50 mg/mL
<b>2</b>	1.5 $\pm$ 0.6	1.9 $\pm$ 0.8	1.4 $\pm$ 0.3	1.6 $\pm$ 0.5
<b>3a</b>	1.8 $\pm$ 0.1	2.1 $\pm$ 0.30	1.9 $\pm$ 0.24	2.4 $\pm$ 0.29
<b>3b</b>	1.5 $\pm$ 0.2	1.8 $\pm$ 0.5	2.0 $\pm$ 0.12	2.4 $\pm$ 0.12
<b>3c</b>	1.4 $\pm$ 0.12	1.7 $\pm$ 0.6	1.3 $\pm$ 0.21	1.8 $\pm$ 0.6
<b>3d</b>	1.6 $\pm$ 0.2	2.1 $\pm$ 0.4	1.5 $\pm$ 0.12	2.2 $\pm$ 0.24
<b>3e</b>	1.7 $\pm$ 0.23	2.2 $\pm$ 0.5	1.6 $\pm$ 0.2	2.1 $\pm$ 0.21
<b>3f</b>	1.4 $\pm$ 0.22	1.8 $\pm$ 0.21	1.3 $\pm$ 0.21	2.1 $\pm$ 0.32
<b>3g</b>	1.8 $\pm$ 0.25	2.2 $\pm$ 0.20	1.6 $\pm$ 0.31	2.2 $\pm$ 0.31
<b>3h</b>	1.5 $\pm$ 0.12	1.9 $\pm$ 0.21	1.6 $\pm$ 0.23	2.3 $\pm$ 0.21
<b>Streptomycin</b>	1.8 $\pm$ 0.25	2.6 $\pm$ 0.28	2.1 $\pm$ 0.32	2.5 $\pm$ 0.34

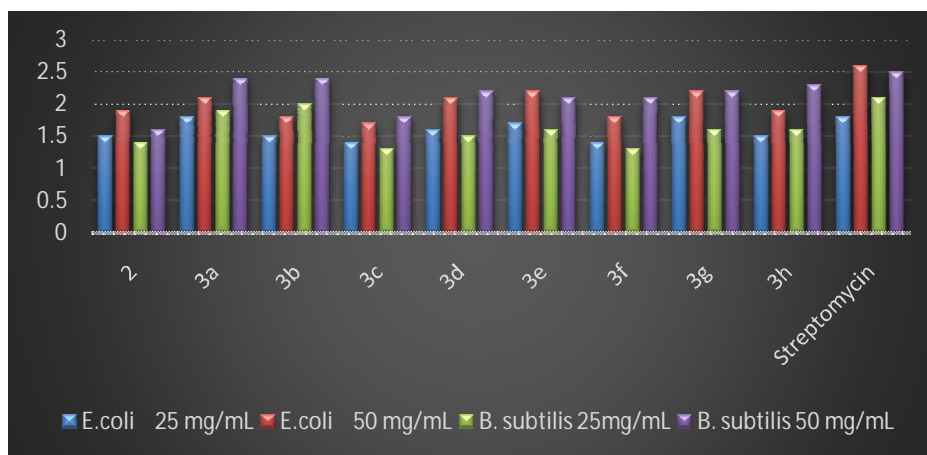


Fig. 1. Antibacterial activity of Compounds 2 & 3(a-h)

Table 2. Antifungal activity

Compounds	Fungi			
	<i>A. flavus</i>		<i>C. albicans</i>	
	25 mg/mL	50 mg/mL	25 mg/mL	50 mg/mL
2	25	53	36	46
3a	44	74	38	78
3b	44	70	55	64
3c	24	69	35	71
3d	36	72	38	73
3e	37	69	39	71
3f	25	67	29	65
3g	38	70	37	69
3h	41	71	38	75
Fluconazole	38	77	39	81

\*Values are represented as the mean  $\pm$  SEM.

\*Values are significant for the standard at 0.005 level of significance.

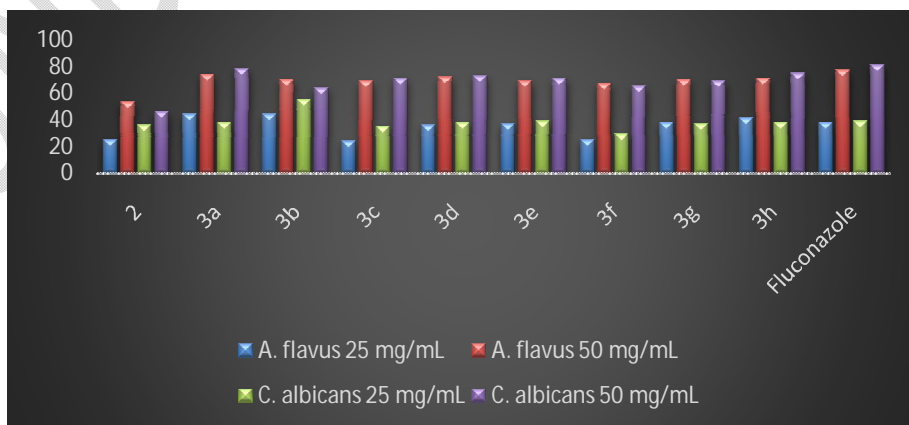
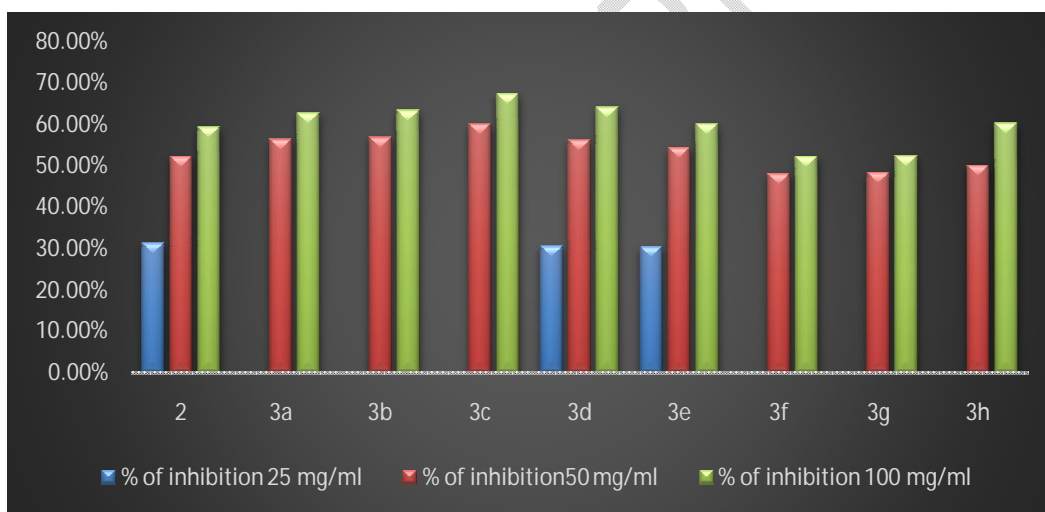


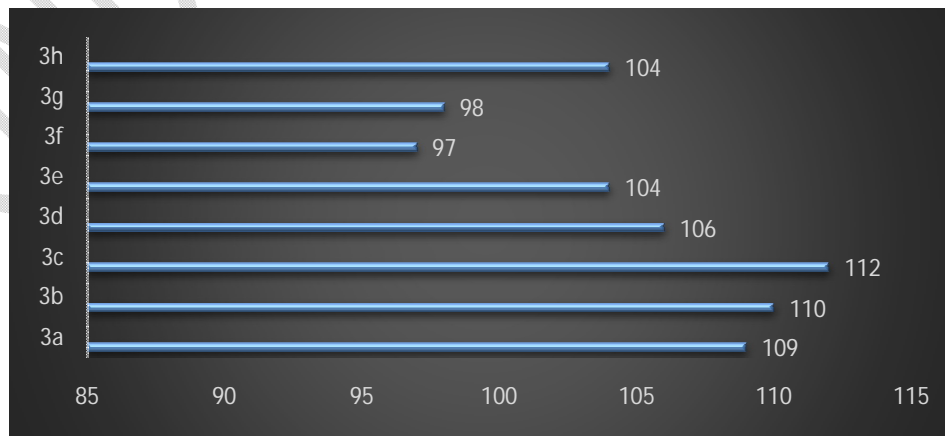
Fig. 2. Antifungal activity of Compounds 2 & 3(a-h)

**Table 3. Antitumor activity**

compounds	% of inhibition 25 mg/ml	% of inhibition 50 mg/ml	% of inhibition 100 mg/ml	IC 50 Value
<b>2</b>	31.4 %	52.2 %	59.4%	96
<b>3a</b>	32.6 %	56.4 %	62.8 %	109
<b>3b</b>	30.4 %	56.9 %	63.4 %	110
<b>3c</b>	34.4 %	60.1 %	67.3 %	112
<b>3d</b>	30.6 %	56.2%	64.2 %	106
<b>3e</b>	30.4 %	54.4 %	60.1 %	104
<b>3f</b>	29.6%	48.1 %	52.3 %	97
<b>3g</b>	30.4%	48.3 %	52.4 %	98
<b>3h</b>	30.6%	50.1 %	60.3 %	104
Std			76.4 %	136

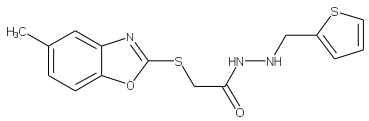
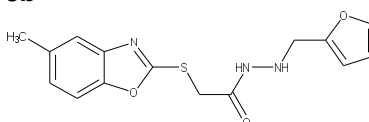
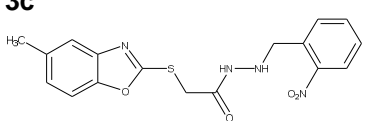
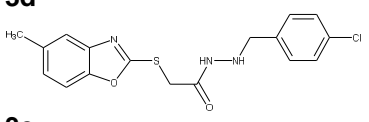
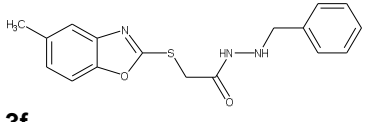
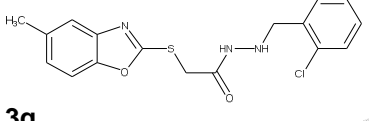
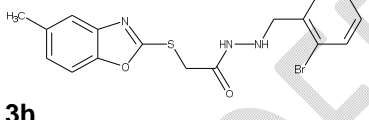
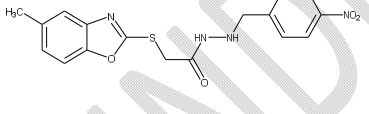


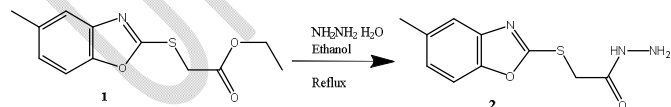
**Fig. 3. Antitumor activity of Compounds 2 & 3(a-h)**



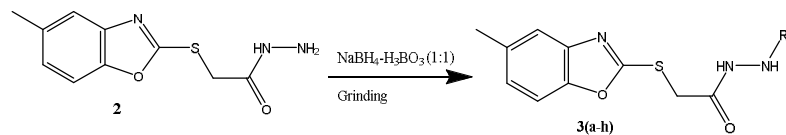
**Fig. 4. IC 50 value of Compounds 2 & 3(a-h)**

**Table 4. Physical data of compounds 3(a-h)**

Compound	Molecular formula	Molecular weight	M.P (°C)
<b>3a</b> 	C <sub>15</sub> H <sub>15</sub> N <sub>3</sub> O <sub>2</sub> S <sub>2</sub>	333.42	235-236
<b>3b</b> 	C <sub>15</sub> H <sub>15</sub> N <sub>3</sub> O <sub>3</sub> S	317.36	250-253
<b>3c</b> 	C <sub>17</sub> H <sub>16</sub> N <sub>4</sub> O <sub>4</sub> S	372.40	296-298
<b>3d</b> 	C <sub>17</sub> H <sub>16</sub> ClN <sub>3</sub> O <sub>2</sub> S	361.84	280-282
<b>3e</b> 	C <sub>17</sub> H <sub>17</sub> N <sub>3</sub> O <sub>2</sub> S	327.40	230-232
<b>3f</b> 	C <sub>17</sub> H <sub>16</sub> ClN <sub>3</sub> O <sub>2</sub> S	361.84	285-287
<b>3g</b> 	C <sub>17</sub> H <sub>16</sub> BrN <sub>3</sub> O <sub>2</sub> S	406.30	298-299
<b>3h</b> 	C <sub>17</sub> H <sub>16</sub> N <sub>4</sub> O <sub>4</sub> S	372.40	280-281



**Fig. 5. Synthetic route for the synthesis of Compound 2**



**Fig. 6. Synthetic route for the synthesis of Compounds 3(a-h)**

### 3. RESULTS AND DISCUSSIONS

#### 3.1 Chemistry

2-[(5-Methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide derivatives were prepared from solvent free reductive amination reaction with different aldehydes. The compound Methyl-[(5-methyl-1,3-benzoxazol-2-yl) sulfanyl] acetate 1 was used to prepare [9], further the target molecules 2(a-h) were synthesized using the intermediate 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide 2. The compound 1 was treated with hydrazine hydrate (0.22 mL, 0.0047 mol, 1.2 eq). The reaction mixture was stirred under reflux in absolute alcohol for 4 hour. Progress of the reaction was confirmed by TLC (Chloroform: Methanol in 7:3 ratio). Reaction mixture was then poured to ice water to obtain compound 2.

The compounds 2(a-h) were prepared by treating 2-[(5-methyl-1,3-benzoxazol-2-yl)sulfanyl] acetohydrazide amine (1mmol) with different substituted aldehydes (1 mmol) for 15 to 20 minutes in an agate mortar and pestle at room temperature (25°C) under solvent free conditions. To the resulting mixture was added 1:1 ratio of Sodium borohydride and boric acid was added and then the mixture was ground for 20-30 minutes until TLC showed complete disappearance of the aldehyde. The reaction mixture was washed with water and further purified by recrystallization.

In the <sup>1</sup>H NMR of 2a, the disappearance of -NH<sub>2</sub> proton and appearance of -NH supported the formation of product. The mass peak of compounds 3a showed at M+ 333 and for compound 3b M+ 317 which matches their molecular weights. The construction of compounds 3(c-h) followed a similar method of preparation.

The compounds 3(a-h) were screened for antimicrobial and antitumor activities. The compounds 3a, 3b and 3g Showed highest degree of inhibition against *A. flavus* and compounds 3a, 3b, 3d and 3h Showed highest degree of inhibition against *C. albicans*, compounds 3a, 3e and 3g has shown the very good antibacterial activity against *E. coli* and compounds 3a, 3b and 3h shows remarkable activity against *B. subtilis*. Compounds 3a, 3b and 3c exhibited potential antitumor activity.

#### 4. CONCLUSION

The overall study reports the synthesis of different compounds via solvent free methods in good yield. The target molecules were characterized and confirmed by mass, IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR analysis and screened for antitumor and antimicrobial activities. The expected target compounds were prepared, structurally confirmed by IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR and mass spectral analysis and *in-vitro* screened for their biological activities. The data described here in indicates that compound 3a, 3b and 3g has developed as potentially active compounds. These molecules have shown significant outcomes as compared to standard drug and considered as potential molecules for further study.

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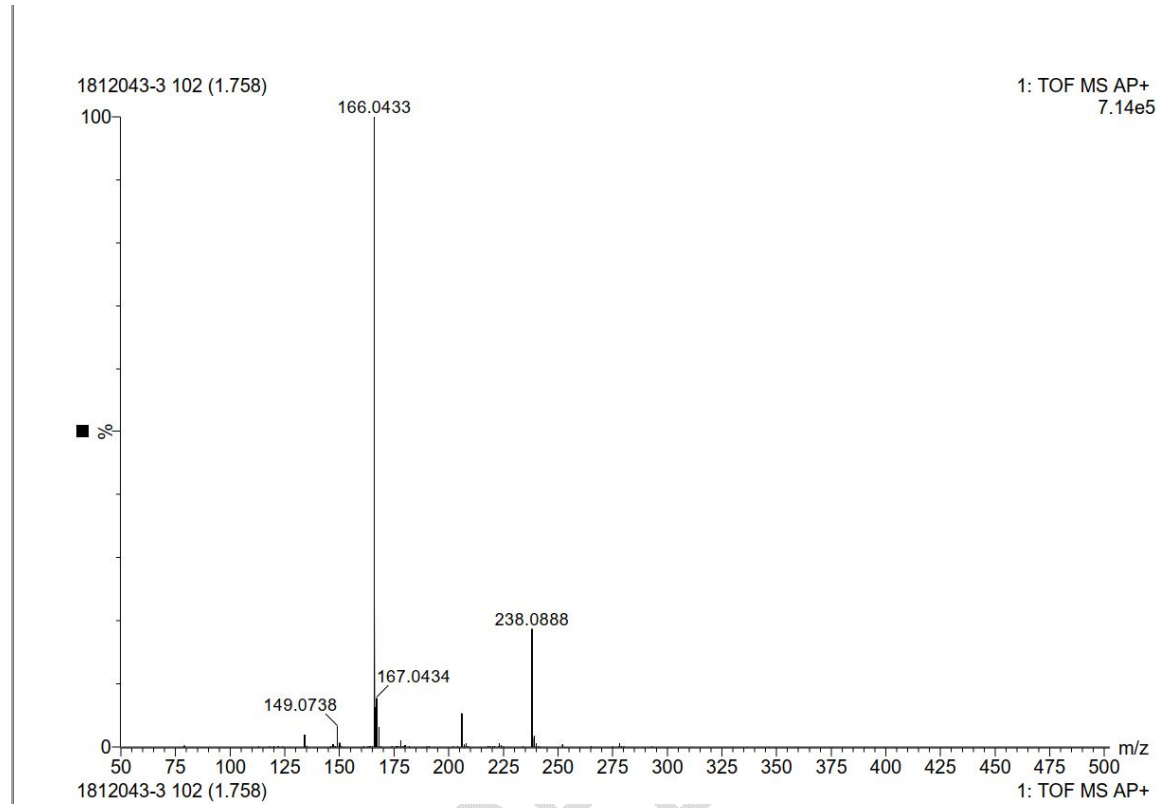
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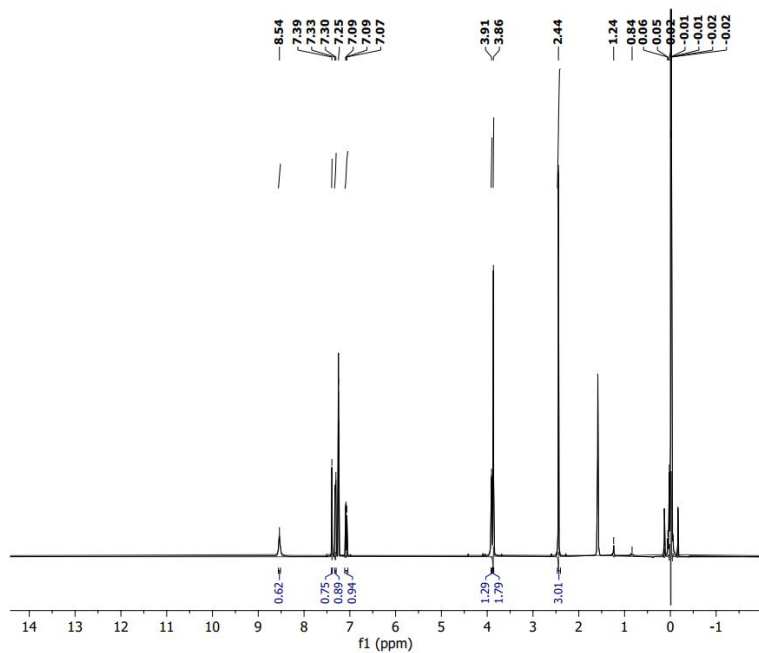
COMPOUND 2

Mass



UNDER REVIEW

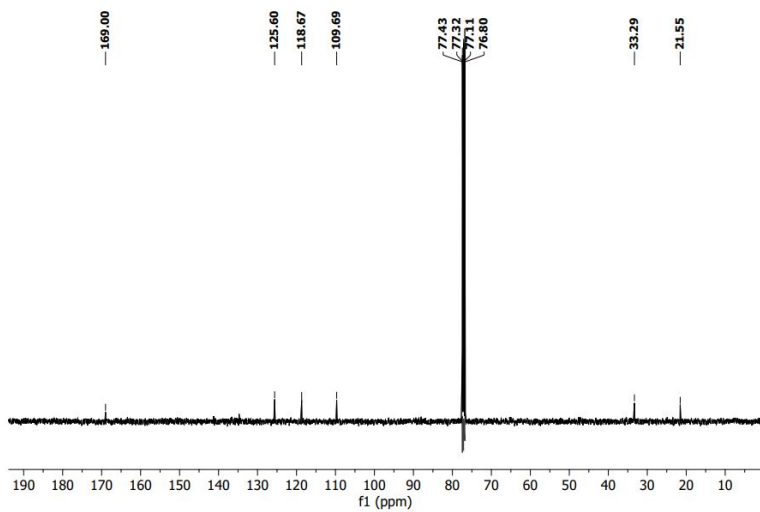
# <sup>1</sup>H NMR



Parameters	
Parameter	Value
1 Title	PJ/3A
2 Comment	single_pulse
3 Origin	JEOL
4 Owner	
5 Site	
6 Instrument	ECA
7 Author	SAIFKUD
8 Solvent	CHLOROFORM-D
9 Temperature	18.2
10 Pulse Sequence	proton.jsp
11 Experiment	1D
12 Probe	2756
13 Number of Scans	32
14 Receiver Gain	56.0
15 Relaxation Delay	5.0000
16 Pulse Width	5.1300
17 Presaturation Frequency	
18 Acquisition Time	1.6358
19 Acquisition Date	2021-02-22T19:19:35
20 Modification Date	2021-02-22T19:23:16
21 Class	
22 Spectrometer Frequency	399.83
23 Spectral Width	8012.8
24 Lowest Frequency	-2007.2
25 Nucleus	1H
26 Acquired Size	16384
27 Spectral Size	52430
28 Digital Resolution	0.15

UNDER PEER

<sup>13</sup>C NMR

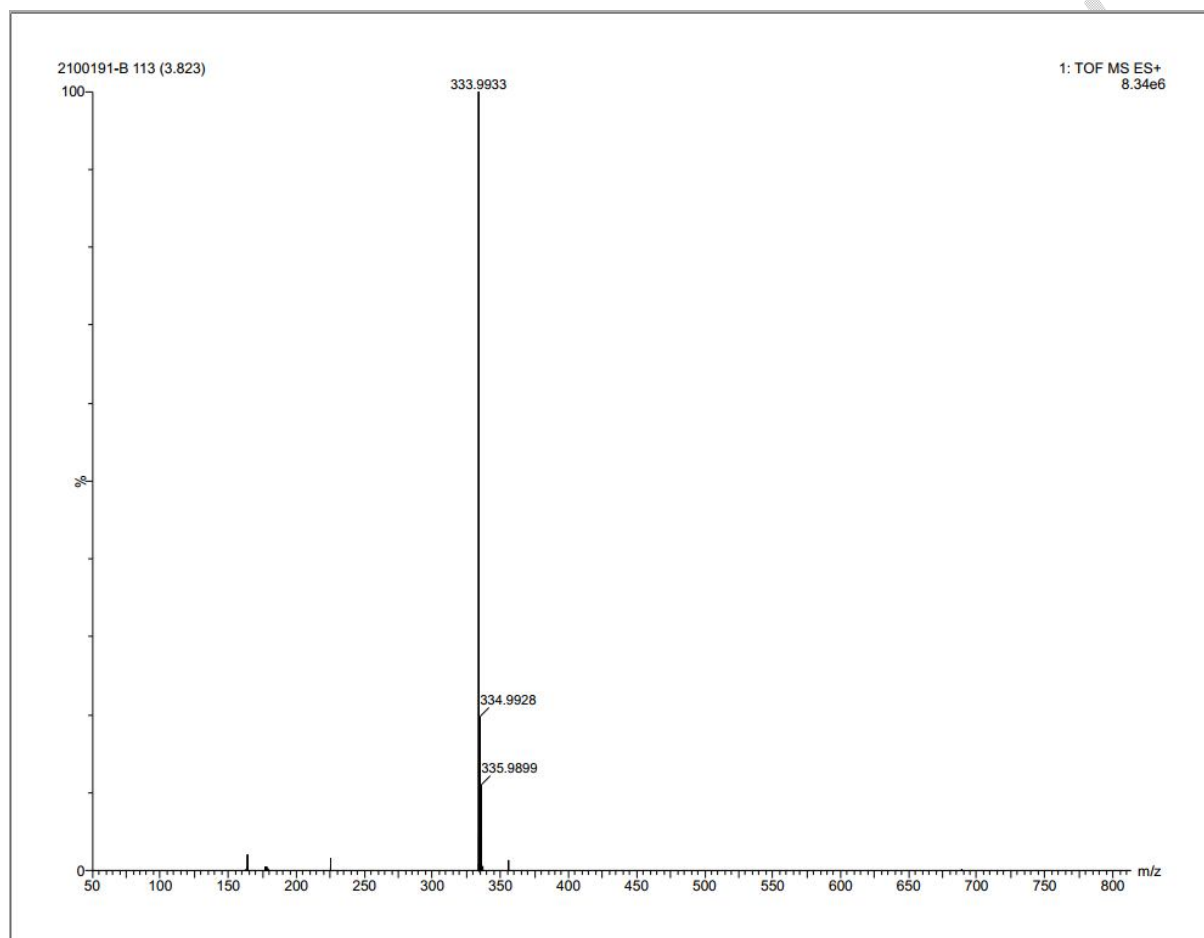


Parameters		
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5	Site	
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7	Author	SAIFKUD
8	Solvent	CHLOROFORM-D
9	Temperature	18.4
10	Pulse Sequence	carbon.jxp
11	Experiment	1D
12	Probe	2756
13	Number of Scans	512
14	Receiver Gain	50.0
15	Relaxation Delay	2.0000
16	Pulse Width	3.8833
17	Presaturation Frequency	
18	Acquisition Time	1.0381
19	Acquisition Date	2021-02-22T19:23:23
20	Modification Date	2021-02-22T19:49:34
21	Class	
22	Spectrometer Frequency	100.54
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25	Nucleus	13C
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27	Spectral Size	52430
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UNDER PEE

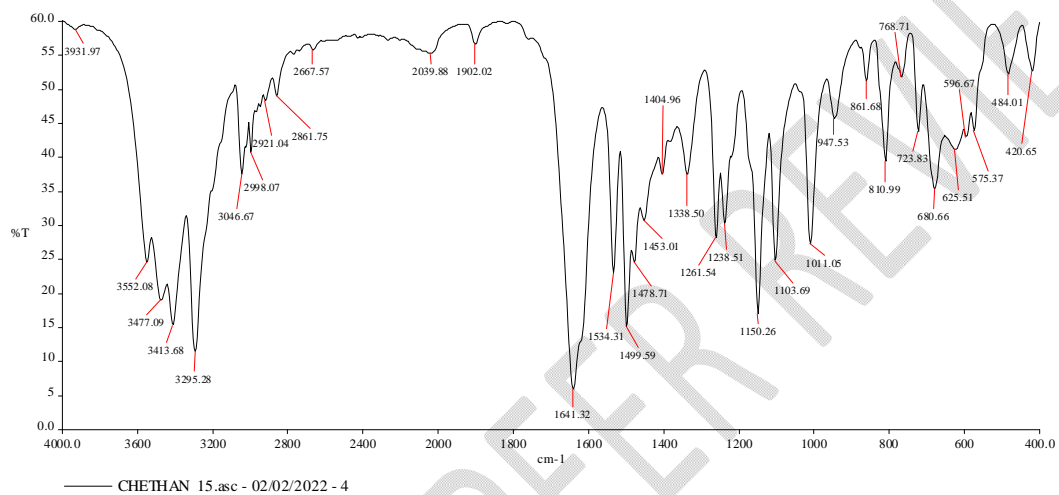
# COMPOUND 3a

Mass

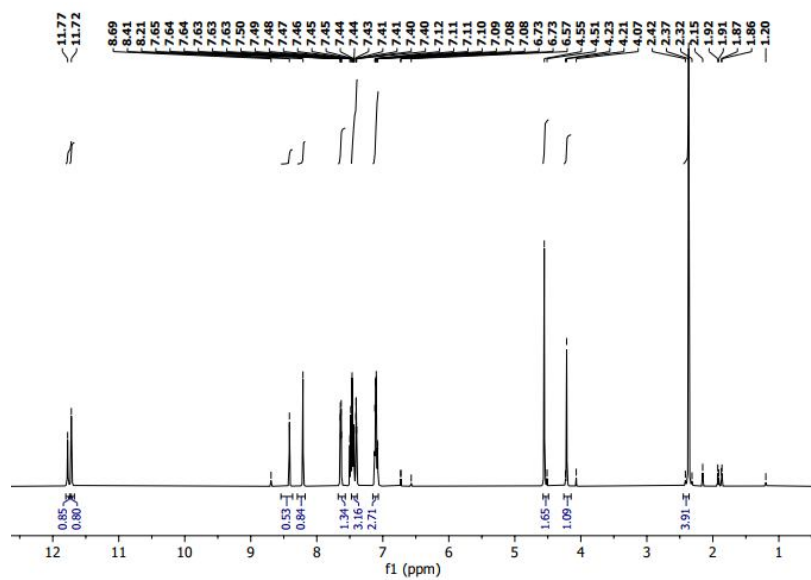


UML

IR



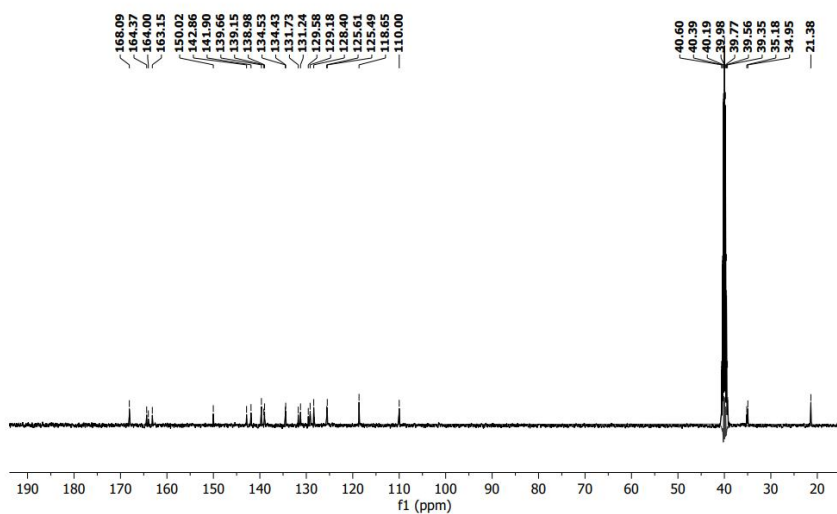
# <sup>1</sup>H NMR



Parameters		
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4	Owner	
5	Site	
6	Instrument	nmr5
7	Author	
8	Solvent	dms0
9	Temperature	80.0
10	Pulse Sequence	s2pul
11	Experiment	1D
12	Probe	ATB
13	Number of Scans	16
14	Receiver Gain	24
15	Relaxation Delay	2.0000
16	Pulse Width	5.4500
17	Presaturation Frequency	
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19	Acquisition Date	2021-02-15T18:27:30
20	Modification Date	2021-02-16T11:43:04
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22	Spectrometer Frequency	399.83
23	Spectral Width	10416.7
24	Lowest Frequency	-1609.9
25	Nucleus	1H
26	Acquired Size	32768
27	Spectral Size	65536
28	Digital Resolution	0.16

UNDER PEE

<sup>13</sup>C NMR



Parameters		
Parameter		Value
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2	Comment	B1-13C
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4	Owner	
5	Site	
6	Instrument	nmrs
7	Author	
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9	Temperature	80.0
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11	Experiment	1D
12	Probe	ATB
13	Number of Scans	756
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15	Relaxation Delay	1.0000
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18	Acquisition Time	1.0224
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24	Lowest Frequency	-1935.6
25	Nucleus	13C
26	Acquired Size	32768
27	Spectral Size	65536
28	Digital Resolution	0.49

UNDER PEER